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Arsenic and mercury concentrations in major landscape components of an intensively cultivated watershed

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"Capsule": *Only fish samples contained arsenic and mercury at levels above published ranges for non-contamination.*

Abstract

To provide an understanding of arsenic (As) and mercury (Hg) concentrations in soil, sediment, water, and fish tissues, samples were collected from a Mississippi River alluvial floodplain located in northwest Mississippi. As concentrations increased approximately an order of magnitude from water (5.12 µg/l) to fish tissues (36.99 µg/kg) and an additional two orders of magnitude in soils, lake sediments, and wetland sediments (5728, 5614, and 6746 µg/kg), respectively. Average Hg concentrations in water, soils, lake sediments, and fish were 2 16 µg/l, 55 1, 14.5 and 125 µg/kg, respectively As and Hg concentrations were within published ranges for uncontaminated soil, water, and sediments. As concentrations represented a low risk Hg concentrations were also low but showed a greater tendency to concentrate in fish tissue The dominant mode of entry of these materials into aquatic systems is through storm-generated runoff Since both metals accompany sediments, agricultural conservation practices such as reduced tillage, buffer riparian strips, and bordering sediment ponds or drainage wetlands will minimize watershed input to aquatic systems. Crown Copyright ⓒ 2000 Published by Elsevier Science Ltd All rights reserved

Keywords Arsenic, Mercury, Watershed, Moon Lake, Mississippi

1. Introduction

Arsenic (As) and mercury (Hg) were used extensively in US agriculture for more than a century from the 1860s to the 1970s (Moore and Ramamoorthy, 1984). The arsenical insecticide, Paris Green, was discovered in 1865 and used as a control agent for the Colorado potato beetle (*Leptinotarsa decemlineata*) (Moore and Ramamoorthy, 1984) Two other arsenicals, lead arsenate and calcium arsenate, were in common use by the early 1900s and were quite effective on a variety of insects including the gypsy moth (*Lymantria dispar*), cotton boll weevil (*Anthonomus grandis*), and orchard pests (Baudo et al , 1990). Use of As as an insecticide declined after World War II with the introduction of synthetic chemicals By the 1970s, environmental regulations in the United States had limited the use of arsenicals to desiccants, fungicide for grapes (*Vitis vinifera*), and an active

ingredient in selective herbicides, especially in cotton (*Gossypium hirsutum* L.) production.

Hg was used as a fungicide/seed treatment for nearly a century although its use in the United States was discontinued due to its toxic properties. Smart (1968) reported that in the 1960s more than 2100 metric tons of Hg was used worldwide per year in agriculture, with Japan alone using 1600 metric tons. Vostal (1972) stated that the amount of Hg used in US agriculture decreased by 10% in 1968, 22% in 1969, and 33 percent in 1970 as the result of public awareness of Hg environmental hazards.

Since As and Hg are elemental compounds, they do not degrade as do synthetic pesticides (Rand, 1995). Thus, their persistence and accumulation can lead to localized or generalized contamination problems in watersheds. Historical and continuing contamination from As and Hg and environmental fate of these materials are of concern because of their toxic properties (Rand and Petrocelli, 1985).

As with many metals, As toxicity varies with its state of valency. In mammals, arsenates inhibit ATP synthesis by

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replacing the phosphoryl group and uncoupling oxidative phosphorylation. Arsenites inhibit thio-dependent enzymes in body tissue proteins such as keratin disulfides in hair, nails, and skin. As does not readily accumulate in most freshwater or marine fishes. There are, however, some marine species such as marlin, cod, and sculpin that may be unfit for human consumption because they maintain relatively high concentrations of As (Moore and Ramamoorthy, 1984). Hg is both an enzyme and protein inhibitor, and strongly associates with suspended solids in natural waters. Hg compounds are highly toxic to plants, and concentrations in plant tissue increase with age. Most Hg found in fish tissue is in a methylated form, which enters fish tissue at a faster rate than inorganic forms. Although fish absorb some Hg directly from water, contaminated sediment and algae are major contributors for herbivorous invertebrates and fish. While Hg is not presently considered to be a carcinogen in mammals, it interferes with DNA replication and displays cytological effects in both plants and animals. Methylmercury crosses the placenta, and several cases of embryotoxicity and teratogenesis have been reported (Moore and Ramamoorthy, 1984).

Although their uses are now restricted, both metals are widely distributed in the environment. Concentrations of 5 mg/kg (5000 ppb) of As are commonly found in soils but may not present a hazard (Edwards, 1973). Information on the occurrence of As residues in municipal water supplies indicates little or no contamination, but plankton, shellfish, and finfish have been reported to have up to 20–40 mg/kg (20,000–40,000 ppb; Edwards, 1973; Price, 1976). As accumulation in soil can be detrimental to soil fauna and certain crops, and As may be translocated into plant tissues. Hg, like As, is persistent and relatively toxic to aquatic biota; Federal Food and Drug Administration guidelines limit Hg in commercial fish to 1 mg/kg (1000 ppb; Marshall et al , 1981)

Regions of intensive rowcrop agriculture found in alluvial valleys and floodplains are high risk areas for contamination from As and Hg because they have historically had high commercial inputs for pest management in addition to natural accumulations from accumulated sediments, soil weathering, and atmospheric deposition. As part of a watershed-scale water quality study, the overall objective of this research was to provide definitive information on concentrations of As and Hg in major components of a Mississippi River alluvial floodplain watershed. Accompanying this objective was the need to provide a basic understanding of surface water and harvestable fish concentrations and to recommend future actions to reduce concentrations of these metals in aquatic systems. These data may then be used in conjunction with additional toxicity data to help determine whether these systems are at risk, or if these materials have potential to cause an adverse effect on aquatic biota.

2. Materials and methods

2 1. Study area

Moon Lake is a large (10.1 km^2) oxbow lake located in Coahoma County, MS, USA. It is one of many naturally formed lakes created over geologic time as the Mississippi River changed meander paths, and it is part of a large fertile river alluvium comprised of thousands of oxbow lakes, river cutoffs, and sloughs (Fig 1) The lake was subject to periodic flooding and flushing by the Mississippi River until completion of the mainline river flood protection levee in the 1920s This levee now completely separates Moon Lake from the Mississippi River. Since permanent settlement began in the area in the 1830s, the percentage of the lake's watershed covered by bottomland hardwoods and natural wetlands has steadily declined in favor of intensive cultivation of cotton, and more recently soybeans (*Glycine max*) and rice (*Oryza sativa*). This increase in agricultural practices has resulted in a concomitant increase in pesticide usage. Over the years, applied pesticides have included As- and Hg-based compounds

Numerous wetlands have been drained and cleared for agriculture in the last three decades, and cultivated land currently occupies over 70% of this watershed Almost all of the 166-km^2 watershed is drained by Phillips Bayou (Fig. 1), a sluggish stream flowing south from Tunica County, MS, through a series of relic
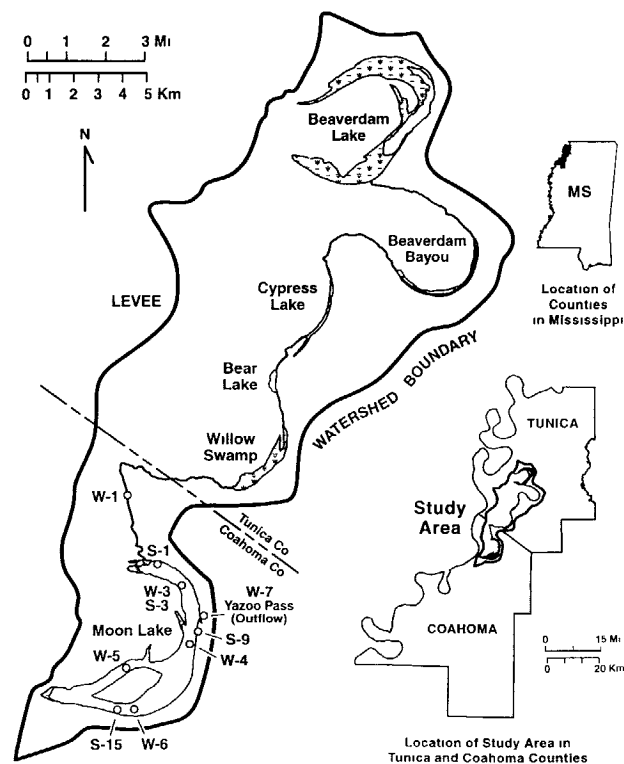

Fig 1 Aqueous (W) and sediment (S) collection sites from Moon Lake, MS, USA

oxbow lakes and associated wetlands Phillips Bayou enters the northern end of Moon Lake Yazoo Pass, the outflow of Moon Lake, breaches the natural levee on the lake's outside perimeter, about one-third the distance around the oxbow-shaped lake.

2 2 Sampling procedures

Surface water samples for As and Hg analysis were collected bi-weekly to monthly from six sites [inflow (Phillips Bayou), outflow (Yazoo Pass), and four sites in Moon Lake proper (Fig. 1)]. Aqueous samples were collected in acetone-and-hexane-rinsed glass jars with teflon lids, returned to the laboratory, and acidified to a pH < 2 with concentrated HNO_3 Samples were stored at 4°C until analysis was performed.

Sediment samples were collected from the lake bottom via a 75-mm diameter core sampler. Eight cores were collected randomly from each of four representative Moon Lake sites from the top 1000-mm of sedimented materials. These were divided into 100-mm increments and were composited for radioactive dating and metal analysis. Sediment samples were also collected from 17 additional sites in the Phillips Bayou drainage network, including various smaller lakes, wetlands, and stream sections. These samples were only collected to 300-mm depths.

Terrestrial soils within the watershed were collected with a 20-mm diameter soil sampling tube. Soil composites for each sample consisted of 20 cores collected to a depth of 200 mm. Samples were collected in the northwest quarter of each square mile of the Moon Lake watershed (miles were used because topographic grid maps are in English units). All sediment and soil samples were collected in acetone-and-hexane rinsed glass jars with teflon lids.

Fish were collected in gill nets at 10 sites representative of lake habitat, dissected where necessary, wrapped in aluminum foil and returned to the laboratory where they were frozen. Fish were sorted according to trophic status (filter feeders, omnivores, piscivores, and benthivores) for analysis. Prey fish, typically eaten whole by other fishes, were analyzed as whole fish. 'Game' fish such as bass, buffalo, catfish, and carp were analyzed as separate flesh and viscera samples.

2.3. Sample analysis

All metal analyses were performed under a cooperative agreement by the Soil–Plant Analysis Laboratory at Northeast Louisiana University, Monroe, LA, USA. Upon delivery to the laboratory, sediment and soil samples were air-dried, finely ground, mixed thoroughly and subsampled. Biological samples were homogenized and subsampled for analysis. For water samples, all metal concentrations were reported in

microgram of metal per liter, and for biological and sediment samples, metal concentrations were reported in microgram per kilogram of sample (ppb) on a dry weight basis Determination of Hg in water was by cold vapor atomic absorption spectrometry (EPA Method 245.1) with a detection limit of 0.2 µg/l. Hg in sediments and fish tissues followed EPA Method 245.5 and 245.6, both with detection limits of 0.2 µg/kg. As concentrations in sediments, fish, and water were determined by stabilized temperature graphite furnace AA spectrometry (EPA Method 200.9 — detection limit of 0 5 µg/ kg for sediment and fish tissues, 0.5 µg/l for water).

2.4. Statistical analysis

Average concentrations of As and Hg were determined for each of the sites sampled. These data were then used to determine whether differences existed between metal concentrations at the different sites. A one-way analysis of variance (ANOVA) was used to test the hypothesis that concentrations of As and Hg at the individual sites within a specific system compartment (i e aqueous, sediment, and fish) did not differ from each other. Differences were considered significant at $P > 0.05$.

3. Results

As concentrations were detected in all watershed components sampled. Average As concentration in water was 5.12 µg/l. Concentrations increased an order of magnitude in fish tissues to 36.99 µg/kg, and an additional two orders of magnitude to 5727 88, 5614.41, and 6746.47 µg/kg in soils, lake sediments, and wetland sediments, respectively (Table 1). Hg was detected routinely in all components measured except wetland sediments. The average Hg concentration in water samples was 2.16 µg/l, while average concentrations in soils and lake sediments increased an order of magnitude to 55.10 and 14 51 µg/kg, respectively; however, no Hg was detected in any of the wetland sediment samples. The average concentration of Hg in fish was 124.9 µg/kg (Table 1).

Table 1
Arsenic and mercury mean concentrations per sample type

Sample type	Season	Arsenic	Conc	Mercury	No of samples
Soil	Overall	5727 88	µg/kg	55 10	61
Wetland sediment	Overall	6746 47	µg/kg	nd	17
Lake sediment	Overall	5614 41	µg/kg	14 51	31
Lake water	Overall	5 12	µg/l	2 16	180/205
Fish	Overall	36 99	µg/kg	124 90	60

Concentrations of As in surface water ranged from 4 48 to 5.99 µg/l. Site 1 on Phillips Bayou inflow had higher concentrations of As than any of the lake sites; however, these differences were not significant (Table 2). Average concentrations of Hg in surface water were slightly lower than As concentrations and ranged from 0 5 to 3.24 µg/l. There were no significant differences in Hg concentrations between any of the surface water sites (Table 2).

Sediment concentrations of As and Hg at different depths for each of the four lake sites analyzed are reported in Figs. 2 and 3. Average concentrations of As in lake and wetland sediments were 5614.41 (±5254.43) and 6746.47 (±2383 04) µg/kg, respectively. In lake (to 100 cm depth) and wetland (to 30 cm depth) sediments, concentrations ranged from 2027 to 8104 µg/kg and 5488 to 7386.67 µg/kg, respectively, and gradually declined in deeper sediments (Figs. 4 and 5). The average concentration of Hg in lake sediments was 14 51 (±19.57) µg/kg; however, no Hg was detected in wetland sediments.

Table 2
Average arsenic (As) and mercury (Hg) aqueous concentrations in individual lake sites[a]

Site	As concentration (µg/l)	Hg concentration (µg/l)
W 1 (inflow)	5 99 (3 95)	2 09 (4 47)
W 3	4 97 (2 08)	1 28 (2 08)
W 4	4 48 (2 46)	3.01 (5 03)
W 5	5 55 (2 16)	1 29 (3 17)
W 6	5 86 (2 26)	0 50 (0 97)
W 7 (outflow)	4 62 (2 12)	3 24 (7 79)

[a] Standard deviation in parentheses

In lake sediments, Hg concentrations to 100 cm depth ranged from 6.47 to 23.87 µg/kg, and, although sporadic, showed a general decrease in concentrations in the deeper sediments (Fig. 6).

Average concentrations of As and Hg in fish (overall) were 36.99 (±148.13) and 124.90 (±193.22) µg/kg, respectively. Fish were also divided into feeding groups For As, the highest concentration was measured in the omnivores [105.01 (±198.73) µg/kg]; however, the highest concentration of Hg was detected in the filter feeders [145 (±264.67) µg/kg] (Fig. 7).

4. Discussion

The average As water concentration in Moon Lake samples was 5.12 µg/l and was well below both the current drinking water standard of 50 µg/l (Hering, 1996), and the EPA established water quality criteria for protection of aquatic biota of 100 µg/l (Hellawell, 1988) However, due to increasing concerns of potential for As-related problems in aquatic-receiving systems, current drinking water standards are under scrutiny The World Health Organization (1993) suggested provisional guideline values be established at 10 µg/l for an As limit EPA is considering a range between 2 and 20 µg/l as proposed new criteria (Hering, 1996). The As concentration in this current study is on the lower end of this proposed range, illustrating the relatively low concentration of As associated with lake waters in this watershed. Published concentrations of As in aqueous lake samples from other studies have ranged from 0 15 to 30 13 µg/l (Moore and Ramamoorthy, 1984; Lacayo
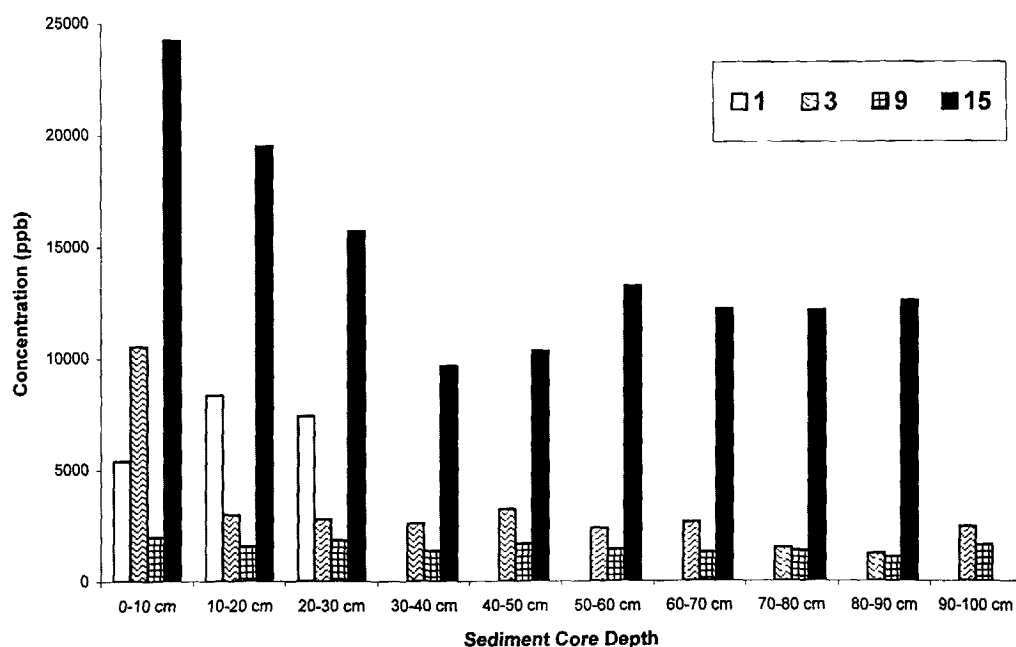

Fig 2 Arsenic concentrations in sediments at different depths at each of the Moon Lake sites sampled
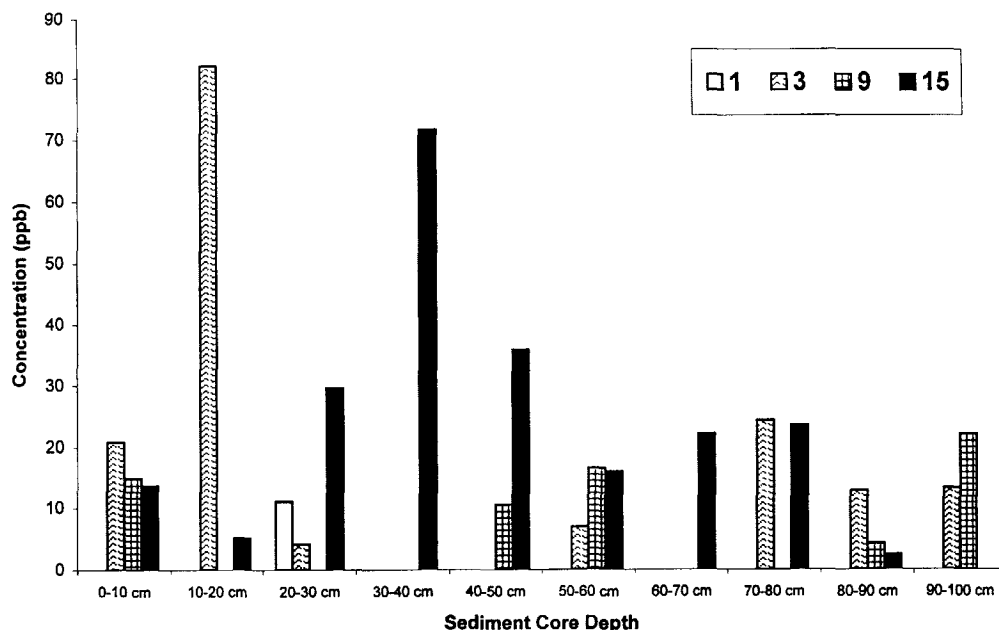
Fig 3   Mercury concentrations in sediments at different depths at each of the Moon Lake sites sampled
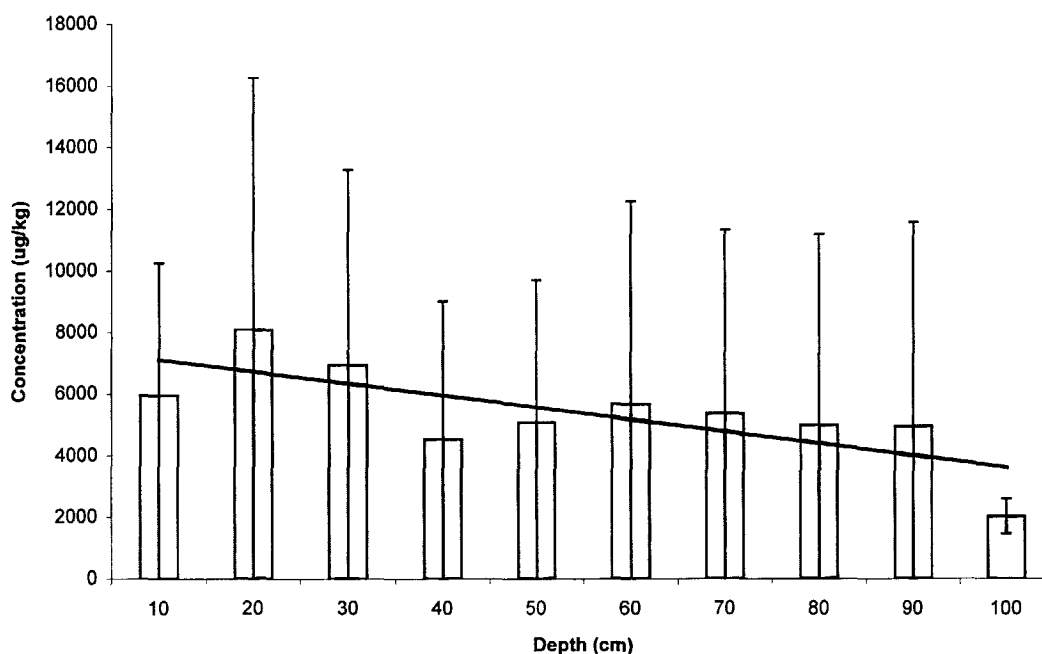

Fig 4   Average arsenic concentrations in sediments at different depths collected from Moon Lake   Trendline represents the decrease in concentrations as depth of sediment increases

et al., 1992); however, aqueous concentrations of As reported to cause toxicity (24–96 h LC50) to fish range from 15,000 to 150,000 µg/l (Sorensen, 1991). The aqueous As concentration in this study was only 5.12 µg/l, indicating a low risk for toxicity to fish in these systems.

As has a higher affinity for fish than overlying water. This is reflected by the increase (an order of magnitude) from water to fish. The average concentration in fish collected from Moon Lake was 36.99 µg/kg. Average

concentrations in fish in uncontaminated waters typically range from 10 to 400 µg/kg (Lacayo et al., 1992) and have been reported as high as 220,000 µg/kg (Moore and Ramamoorthy, 1984) in contaminated waters.

As has a higher affinity for sediments than for either fish or water within aqueous systems (Viraraghavan et al., 1992). In this particular study, concentrations in soils, and in wetland and lake sediments were 5700,
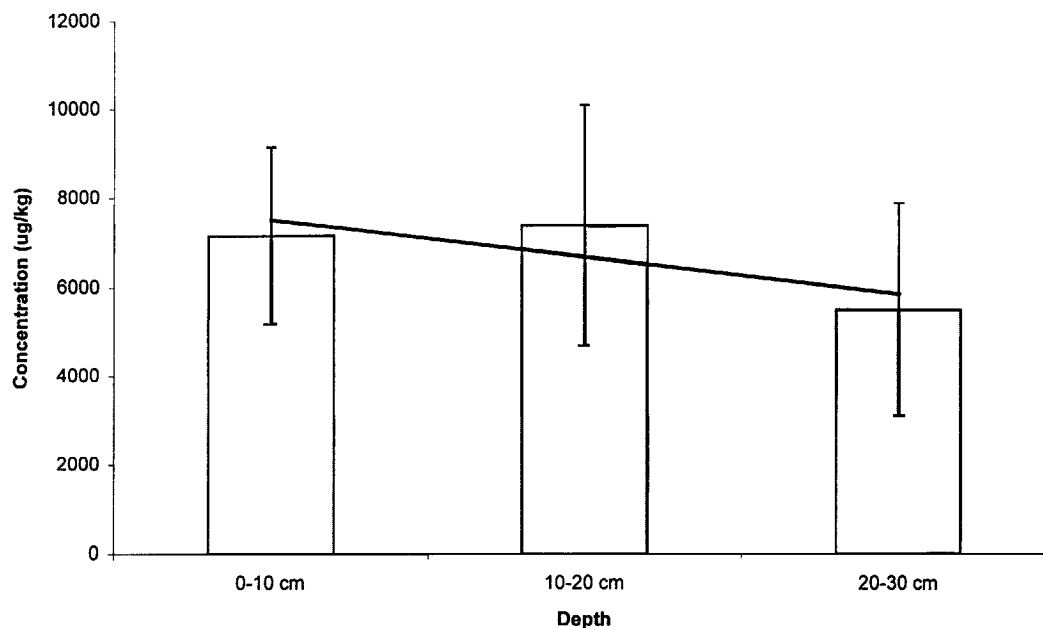
Fig 5 Average arsenic concentrations in sediments at different depths collected from wetlands Trendline represents the decrease in concentrations as depth of sediment increases
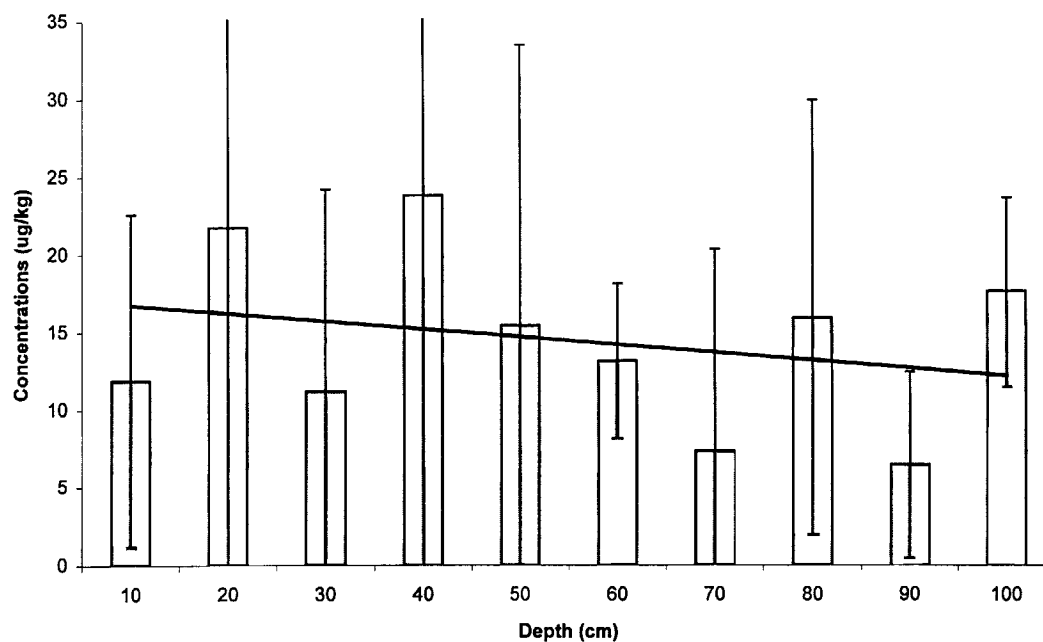

Fig 6 Average mercury concentrations in sediments at different depths collected from Moon Lake Trendline represents the decrease in concentrations as depth of sediment increases

6700 and 5600 µg/kg, respectively. Concentrations in sediments previously uncontaminated with As typically range from 500 to 59,000 µg/kg (Moore and Ramamoorthy, 1984), and uncontaminated soil concentrations have been reported to range between 1000 and 20,000 µg/kg (Lederer and Fensterheim, 1983). Lacayo et al (1992) reported concentrations of As in several lake sediment samples ranging from 2700 to 13,200 µg/kg. Concentrations in this current study were well

within average concentrations reported for uncontaminated sediments throughout the United States

Hg concentrations in all watershed components were less than As concentrations. Threshold concentrations for concern are somewhat ambiguous because of the complexity of Hg toxicity. EPA criteria for protection of aquatic biota is 0.05 µg/l (Hellawell, 1988). For comparison, the EPA established drinking water standard for Hg is 2 µg/l. Food and Drug Administration
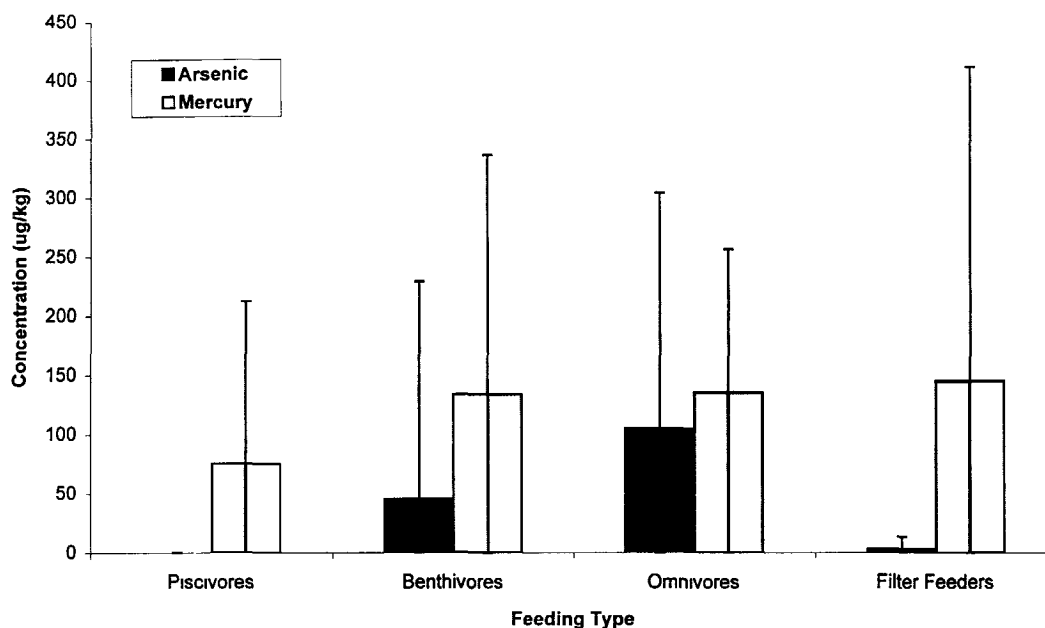
Fig 7   Mean measured concentrations of arsenic and mercury in fish (different feeding types) collected from Moon Lake

guidelines for commercial fish are 1000 μg/kg Hg. The average Hg concentration in Moon Lake water samples was 2.16 μg/l. The range of reported concentrations for 'unpolluted' waters is between 0.02 and 0.1 μg/l (Moore and Ramamoorthy, 1984) Aqueous concentrations of Hg reported to cause toxicity (96-h LC50) to fish range from 11 to 1800 μg/l (Baudo et al., 1990). The aqueous Hg concentration in this study was only 2.16 μg/l, indicating a low risk for toxicity to fish in these systems; however, the value was still slightly above the safe drinking water concentration, and approximately four times the aquatic biota protection criteria.

Concentrations of Hg in fish for this particular study were reported at 124.9 μg/kg dry wt. The criteria for concentrations of Hg in fish is 1000 μg/kg (Moore and Ramamoorthy, 1984), and concentrations have been reported to average between 50 and 2000 μg/kg (Henderson et al., 1972). Rood (1996) reported concentrations in Florida lake bass ranging from 30 to 1380 μg/kg. In this same report, concentrations based on a United States survey of freshwater fish were reported to range from 10 to 370 μg/kg (Rood, 1996). This indicates that concentrations of Hg in fish in Moon Lake are below the national average and are also below EPA established criteria.

Background Hg concentrations in sediments collected from uncontaminated sites typically range between 10 and 200 μg/kg. However, in sites previously contaminated with Hg, concentrations have been reported as high as 2010 mg/kg (Baudo et al., 1990). Hg concentrations in soils and lake sediments presented in this study were 55.1 and 14 5 μg/kg, respectively, and were within the range reported for sediment concentrations in relatively uncontaminated sites. No Hg was detected

in any wetland sediment samples. Low oxygen levels in wetlands, brought about by decomposing plants and higher sulfide concentrations, allow growth of bacteria that convert Hg into methylmercury. Methylmercury is an organic form of the metal that is quite toxic and highly soluble, easily absorbed by plants and animals, and does not sorb readily to sediments within the system.

5. Conclusions

This particular study was designed to measure concentrations of As and Hg associated with different compartments of a northwest Mississippi watershed. The underlying goal was to determine concentrations associated with different system compartments to gain a better understanding of the fate of these materials from a watershed perspective. The ultimate goal of this research is to develop an improved understanding from a watershed perspective in order to provide recommendations that will result in water quality improvement within these watersheds.

The two particular metals investigated in this study were of interest due mainly to their past uses for control of agricultural pests. Both Hg and As were used extensively in the study area; therefore, the obvious source of these metals within this watershed is the soil surrounding the various aquatic systems. With As, it appears that lake and wetland sediments are the dominant sink for this material, with the lowest concentrations observed in the overlying waters Data illustrate the ability of soils and sediments to bind As, resulting in little release to overlying waters. Hg is slightly different than As in that fish, in this study, accumulated it more easily Soils have

the ability to bind this material, but once in aquatic systems, it has a higher affinity for fish than for either wetland or lake sediments.

In this particular study, concentrations of As in all components investigated were within ranges of concentrations reported for uncontaminated water, sediment, and fish. These data allowed a better understanding of As in this watershed and illustrated the low potential for problems arising in this system due to As. Hg concentrations were lower than As concentrations and were all within average levels observed in uncontaminated sites, except for concentrations in water. Aqueous Hg concentrations (average of 2.16 μg/l) only slightly exceeded the EPA drinking water standard of 2 μg/l.

Because the major source of As and Hg entering this watershed is from terrestrial soils, several management practices may be proposed The dominant mode of entry for these metals into aquatic systems is storm-generated runoff. By reducing the amount of sediment entering these systems, we directly reduce the amount of contributed metals. Thus, management practices which reduce runoff, particularly sediment, from watersheds should sufficiently reduce the amount of Hg and As entering the aquatic ecosystem so that Hg falls within strict (drinking water) guidelines and As is further reduced. Reduced tillage systems result in less erosion. Also, systems designed with buffer strips and edge-of-field sediment ponds or drainage wetlands bordering the aquatic drainage will help to minimize erosion and sedimentation in streams and lakes. By keeping the As and Hg from reaching aquatic systems, we can minimize potential impacts that these materials will have within these watersheds and downstream waters.

These watershed component data, coupled with additional toxicity data, would produce necessary information to determine impairment from As and Hg once they enter streams and lakes. This, in turn, will provide a database to realistically pursue effective farm management practices which lessen aquatic contamination within the watershed, while allowing production agriculture to continue with minimum restraints.

6. Disclaimer

Acknowledgements

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